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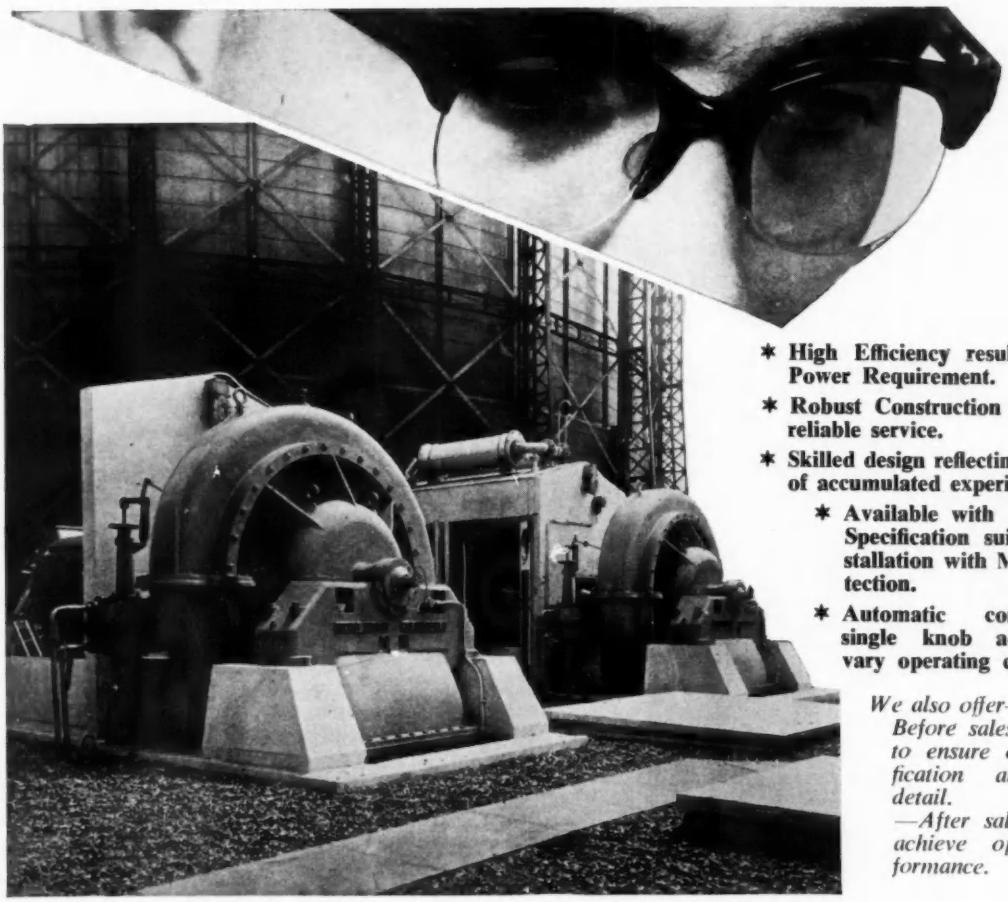

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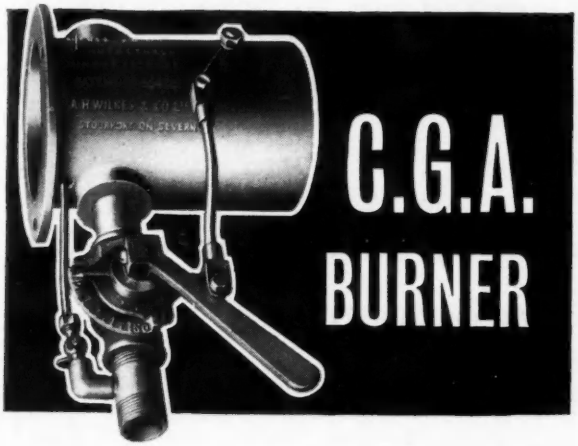

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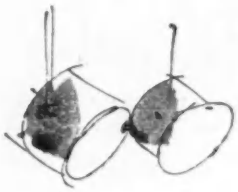

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Gas Journal

COKE OVENS • BY-PRODUCTS • CHEMICALS

Where the chemists come in

THE importance of the organisation behind the engineer is ever increasing, and without the necessary exact figures and statistics to call upon, the top technical executive would find his job far harder, if not quite impossible. The chemist in our industry occupies an important position in this organisation.

Two papers read at the recent research meeting, abstracts of which we are publishing this week, illustrate the type of problem which the chemist has to solve, as the engineer sets about exploiting new processes or modifying existing plant to use new feedstocks which may be less expensive, and more easily available than those he conventionally uses. The sharp increase in the price of gas oil for c.w.g. or oil gasification plants and its consequent replacement in many plants by p.f.d. is an example of this.

The first to be read was by Professor A. L. Roberts and Mr. C. P. Ward, of the University of Leeds; it was called ' Modern Analytical Techniques Applied to the Gas Industry,' and was the first of a series of such papers. It dealt entirely with the analysis of p.f.d. by gas liquid chromatography—G.L.C. in some quarters. The second paper was entitled ' Analysis of Oils for Gas Manufacture,' by Mr. A. B. Densham and Mr. P. A. A. Beale, both of the London Research Station.

These two papers stressed the need for some quick means by which low boiling hydrocarbon distillates and light oils may be tested as a means of ascertaining their value for both pricing and plant efficiency.

Professor Roberts goes into some detail in his explanation of the analysis by gas liquid chromatography and we, who are not normally in a modern analytical laboratory or conversant with the most up-to-date methods, appreciated the detailed points he made.

When we recall that within the last few years Professor Roberts read a paper to the Institution on analysis by infra-red spectroscopy, we begin to realise the extent of this author's mental agility and also the speed with which even research and analytical methods are changing.

Undoubtedly the chief advantage of chromatography is its speed. Once the apparatus has been calibrated the analysis may be carried out in a matter of hours, even when as many as 15 constituents may be involved.

An analysis by more conventional means, fractional distillation for instance, even where possible, would need a far larger sample and take perhaps days. This method is, then, a most useful tool in the analyst's equipment, but like any other ' miracle ' method, its limitations must be recognised and accepted. One of these limitations is its application to quantitative measurements, and the authors show the importance of ' internal normalisation ' in this connection.

Mr. Densham's paper, though concerned with analyses, does not concern itself with any particular method but rather reviews the means available for determining the composition of a hydrocarbon oil to be used as a feedstock, whether it is to approach the standards laid down by the board for the purchase of such oils, and what if any will be the effect of the oil's composition on the working of the plant.

When a sample of a consignment of some size is to be analysed for control purposes, it is very helpful if the delay between sampling and knowing the answer is kept as short as possible. Even when an accurate analytical method has been worked out and adopted, it sometimes happens that a quicker but less accurate means, depending on the easy measurement of some physical characteristic, is in fact more appropriate. Such a method must, however, be reliable and capable of repetition. The availability of such methods was discussed.

The classical method, the Griffith method, originally worked out in 1928 by Dr. R. H. Griffith for the evaluating of oils for the enrichment of c.w.g., the authors find gives too high a figure for paraffins, by some 10%, but it is the only method, say the authors, that gives good correlation with enrichment values over a wide range of oil composition. It is noteworthy that the North Thames Board still uses this method and presumably is satisfied.

The paper discusses a number of variations of the method as well as several that are more modern, and from a table comparing the results from each, the agreement or diversity between them is clearly seen.

We Wish All Our Readers A Happy Christmas

Underground storage

THE chairman of a nationalised industry, or of any industry for that matter, acts wisely when at the earliest opportunity he seeks to take the public into his confidence over any new project which seems vitally to concern their traditional rights and interests. In such circumstances, Sir Henry Jones, Chairman of the Gas Council, visited Winchester last week to discuss with local interests the proposal to store some 10,000 mill. cu.ft. of gas under an area of 3,500 acres in and around Chilcomb. In last week's issue we published brief details of the scheme which, though new to this country, has been used for many years elsewhere —notably in the States, in France, and in the U.S.S.R.

Our industry is fortunate in having a Chairman with such a charm of manner and such an aptitude for handling meetings of this kind that many of the good citizens of England's ancient capital who came convinced that they were threatened with being perched on the top of a volcano went away largely satisfied that the most stringent precautions were being taken to preserve both the safety and the traditional rights and privileges of the individual. Sir Henry was supported at the meeting by so formidable an array of experts that one felt they could have adequately answered questions on any subject from the theory of relativity to the origin of species. As it was, however, the questions followed the not unexpected pattern of such enquiries; and it may be of interest to underline some of the principal points raised.

It should be explained that two distinct areas of storage are involved in the plan—the 'green' zone which forms the main area and the 'red' zone which is in effect an overflow area extending beneath the south-eastern sector of the city. It is this latter which is being most strenuously opposed by the City Council, who do *not* oppose the main project. On this point, Sir Henry emphasised that where human life is concerned there are no relative degrees of safety. Were they not satisfied beyond all reasonable doubt that the whole scheme was absolutely safe the presence of one solitary farmhouse above the storage area would be as indefensible as a whole residential area.

Contamination of public and private water supplies came under question in relation to the presence of water in the lower greensand of the storage area. It was pointed out, however, that water supplies are normally drawn from levels well above that of the lower greensand; and even if the sinking of deep wells in adjacent areas caused any of this water to be drawn away towards the public supplies the rate of carbon monoxide absorption is so extremely small and extremely slow that there is virtually no risk of contamination.

Some anxiety was not unnaturally expressed as to how effectively the geological formation would contain the gas at times of full storage; but assurance was given that an efficient system of 'monitor' shafts round the perimeter of the area would instantly detect the presence of gas where it should not be.

The clause in the Bill which prevented landowners from boring or excavating below sea level over the whole site was questioned; and it was explained that this still enabled boring to quite a considerable depth—deep enough for all practical purposes. This clause was inserted to guard against boring through the gault into the greensand where the gas was to be stored, since it would scarcely be commonsense for the industry to spend considerable sums of money to provide this storage for gas if there was nothing to prevent persons, by accident or design, boring down into it and letting it out again. The rights of existing wells and powers to sink new ones were fully preserved within this limit.

It was inevitable that the question of depreciation of residential property situated over the storage area should come in for discussion; and on this Sir Henry felt it was largely a matter of current opinion. There was obviously no denying that until this entirely new project had been accepted as a normal facet of our industrial plan certain people must feel that property above the area might be less attractive than it was before. This is clearly more of a psychological than an actual objection. Coupled with the fact that scarcely anything would be visible above ground and what little was visible would be most carefully designed to blend with the surroundings, there could be little complaint on this score. Once the public is used to the idea, underground gas storage would doubtless be accepted with as little comment as coal mines (which obviously extend beneath many residential areas), underground water reservoirs, or underground railways.

A representative of the electrical industry naïvely questioned Sir Henry upon the extent to which the pumping plant would rely upon electrical power—bearing in mind such things as power failures. The Chairman doubtless appreciated this solicitous enquiry from a rival industry but assured them that they were relying on diesel engines for their pumps and compressors. He also stated in answer to another question that the presence of this underground gas could have no possible detrimental effect on electrical cables or installations either above or below the surface.

A gas consumer—and it is nice to feel that they too are concerned, though one would hardly have thought so from the general trend of the discussion—was worried whether the gas would suffer as a result of water or other impurities being entrained from the storage area. It was made clear that any impurities, whether liquid or solid, brought up with the gas would be effectively eliminated before entering the town's mains. 'The quality of our gas will be of the same impeccable standard to which our consumers are accustomed!'

This meeting at Winchester did much to clear the air and set at rest a good deal of local anxiety. And as Sir Henry Jones pointed out in his closing remarks, the greatest safeguard lies in the fact that the Bill still has to pass through Parliament, where the public interest will be closely watched at every step. We feel the time will be when the citizens of Winchester may well feel proud to have been selected as the guinea pigs for a project which will streamline the gas manufacturing industry of this country.

Gas statistics 'down under'

STATISTICS from gas industries overseas are always of interest, especially outside Europe and America. Gas 'down under' can be particularly so because the conditions under which the industry operates in Australasia are so very different from those in Britain. The comparatively small number of consumers in relation to the size of the country both in Australia and New Zealand must make the development of these countries' gas industries on modern lines a matter of some difficulty, but by making the National Gas Association of Australia responsible for collecting information from both the Australian and the New Zealand gas industries, a clear picture of their activities is immediately available.

That the Australian industry is expanding is shown by the rise in gas made and sold in 1960, this being 8.2% and 6.5% respectively above the 1959 figures. A total figure of gas sent out at 269.26 mill. therms and of sales at 228.74 mill. therms are both records for Australia. Of these sales, 72.4% were domestic, 15.7% industrial and 11.9% commercial. The domestic consumer then is still of prime importance, although as the industrialisation of the country proceeds, the industrial consumption may well grow.

Coke sales have fallen once more, but the total revenue from this source has increased by 2.5% to £5.6 mill. For appliance sales it was also a good year since they showed an increase of 7.7% at £7.67 mill.

In New Zealand, where the gas industry is on a somewhat smaller scale and operating in very close competition with the electrical supply industry, the 26 undertakings which submitted statistics to the Australian Association represent 98.9% of the gas producers, while the New Zealand Association was able to supply figures for gas made and sold from the remainder.

Total gas sent out for the Dominion totalled 25.86 mill. therms, an increase of 1.9% over the previous year and gas sales at 20.76 mill. therms rose by 1.0%.

Though the revenue from gas sales increased by 1.6%, the number of consumers fell by 1.3% to 167,106, and appliance sales dropped by 8.9%. In both countries the number employed by the industry fell over the year, in Australia by 2% and in New Zealand by 3.4%, though in both there was an increase in wages and salaries paid, of 8% and 7.4% respectively.

The consumption of gas per domestic meter for Australia as a whole rose from 135 therms per year in 1959 to 141 therms in 1960; in New Zealand in the same period it increased from 98 to 101 therms. The discovery of natural gas at Kapuni in New Zealand may have a considerable effect on gas consumption in the Dominion.

Natural gas at Kapuni

THE discovery of natural gas at Kapuni in New Zealand is likely to have an epoch-making effect on the country's economy, provided it can be geared to meet the possibilities offered by such a source of cheap fuel and the capital is available to exploit it. The New Zealand gas industry is naturally delighted to learn of the discovery and has expressed its confidence that the capital can be made available for laying the necessary pipelines and the other plant that will be needed to develop the demand for gas throughout the Dominion.

Judging by the difficulties which the industry has had to face in the last few years, largely the result of rising costs of imported feedstocks and labour with a decreasing demand, such a find is indeed good news.

The size of the discovery, however, is not enormous, being equivalent to the thermal demand for a city the size of Greater London, with a population of 8 to 9 million inhabitants.

The great problem facing the Government is whether the deposit is really big enough to develop industry to consume it, or merely to use it as a means of steam raising for electricity generation.

There appear to be vast deposits of iron sands in the country, which might be economically worked provided it can be done on a big enough scale with a cheap source of fuel, and this may be a chance to do so.

The gas itself contains both light petroleum oil and a comparatively high proportion of carbon dioxide. Both of these would have to be removed, the first to provide 50% of the country's motor spirit and the second to upgrade the residual gas. The plant for both operations is expensive.

It is not often that a find of natural gas is responsible for such mixed feelings and doubts as to its potential use, and it is interesting to consider the situation rather more carefully. New Zealand, a country roughly about the size of Britain, has a population of some 3 mill. inhabitants and is principally agricultural. The centres of potentially large gas consumption are the larger towns, which are some distance from each other, and therefore the length of non-productive pipeline is likely to be extensive, without the very large consumption potential as it enters the 'city gates' that is common in the U.S.A.

To burn it on the spot in power stations, or to start a new industry in the neighbourhood of the wells appear to be two possible alternatives, but if the Government decides on either, the New Zealand gas industry will be justifiably disappointed and will rightly consider that a chance for its 'comeback' in a big way has been snatched from beneath its very nose.

A measure of satisfaction

AN encouraging, even rosy, word picture was presented by Mr. R. N. Bruce, Chairman, at the South Eastern Gas Board's Annual Joint Consultative Committees Conference, held at Eastbourne.

Mr. Bruce said that in the last financial year, if all factors had been normal, instead of having a deficit of £460,000, they would have had a surplus of £400,000. Today they had an increase in the sales of gas over the last year, and he prophesied that on March 31, 1962, they would again be able to report record sales of gas.

Referring to the report of the Select Committee, Mr. Bruce said that it was quite kind and congratulatory to their industry; the paragraph he cited could justifiably be so described.

Turning to another exceedingly bright star in the industry's self-contained firmament, the Chairman said that he thought 10 mill. cu.ft. of imported methane a day would be taken by the Board, 8% of the rated maximum production capacity.

The Chief Distribution Engineer, Mr. T. W. Gibson, then told of his side of the Board's activities. He referred to the advance made since vesting in the closing of old gasworks and the integration of the distribution system. This had needed a reorganisation of the distribution department.

Mr. Gibson then reviewed developments at present being undertaken in the conversion of holder stations, and the advances in pipelaying.

This general air of encouragement and satisfaction was slightly disturbed by the comments of the three speakers from the staff side of the Board.

Mr. R. J. Turnbull, said that the sale of appliances was not an end in itself, but a means to an end; their main product was gas. The Select Committee had said that the cost of gas must be reduced; whatever the result of the Gas Council's deliberations on this matter, it was bound to cause considerable upheaval.

Mr. P. C. McNally stated strongly that if they were going to keep pace with modern industry, they must give the proper education to their young people. He believed that the training of the older man should be given considerable attention, and expressed some concern that training was too often viewed as an interruption of work.

A belief in the value of joint consultation was expressed by Mr. F. C. Walker, who said that without it they would suffer from frustration and would not know where their industry was going. If they could avoid disputes they were all contributing to the future of the industry.

Perhaps one could detect a certain satisfaction with the situation as it was described at this conference by the Board Chairman. He explained the situation which led to a deficit last year, and he made reassuring forecasts for the future. This is admirable, provided one does not lean too far away from concern. For one must yet ask whether the industry is in such a position that it may be reassured. Or does it still need fighting talk, and a fighting outlook?

Behind the historic occasion

By W. H. Johnson

BY printing a progress report on the gas industry at regular intervals, *The Financial Times* provides a valuable and solid effort in public relations for which we should all be duly and truly thankful.

It will be seen that the biggest guns are brought to bear upon the target, and the reports are therefore all the more momentous. That contemporary symbol, 'A spokesman for the industry,' too often used as a straw in the wind to test the strength of dissent, has a day off.

In the nature of things one expects, and finds, that the Chairman presents the overall picture of present and future policy, and it is therefore to this that the main attention must be directed.

Disturbing—in parts

After making due allowance for the brevity enforced by the occasion, and for the prospective readership, it is still, in parts, a somewhat disturbing document. If it were a chairman's annual address to a surging Company paying 15% tax free it would be received well, but if followed by a declaration of no dividend it would undoubtedly provoke questions from the floor.

It is doing just that now, with the justification that, according to the theory of the industry's new constitution, everyone of us has a stake and an interest in its wellbeing.

Everyone will welcome the official underlining of the consumer's view, that the only thing wrong with gas is its price, coupled with the linking of selling price to cost of production.

It is not quite so easy to accept the stark black and whiteness of the picture in which every factor contributing to a solution of the cost problem is presented as immediately acceptable and beneficial in its own right.

In particular, I take exception to the statement that the Algerian methane project will give confidence to everyone in the industry and to present and future consumers.

The gas industry must have changed a great deal in the last few years if it can be truthfully claimed that every member is of one mind on matters as important as this—or on any matter. Is this rather an assumption: That people who shelter under one hat can have only one communal head?

Present and future consumers, anyway, are under no obligation to express confidence without conviction, and it would have been safer to leave them out.

They are bound to have awkward ideas about the wisdom of allying a part of our economy to a part of the world which is more politically unstable than most. A part, moreover, which has practised the use of high explosive as a haphazard political argument for some seven or eight years and shows little sign of abandoning it.

It is none too obvious, either, that the coal industry is content to sit back and wait until the gas industry is comfortably settled into a foreign alliance before it considers 'realistically' its new attitude to coal.

Granted that the coal industry has itself fumbled badly in the past, one can sympathise with its view that blood is thicker than water, and there is justification for its excuse that good gas-making coal gets deeper and costlier.

This is where the Lurgi-type plant comes in, or should come in, but in the Development Engineer's report it is hinted, and in the editorial comment stated, that it is economically a bit shaky as a production unit.

What a pity we were not let into the picture a little more about those new plants which are in a raw state at present.

Will they, too, be able to show a profit or a loss depending upon what the cost accountant puts into, or leaves out of, the balance sheet? Anyone who extracted crude benzole from gas before the war will know how flexible the balance can be.

We—the consumers—are in need of some reassurance about real progress on the manufacturing research side of the industry. On the other—the utilisation—side there is no doubt about the technical advances being made both by the industry and by the appliance makers.

But the Lurgi plant is someone else's idea—a foreigner. Was it economical to operate in its own country, and if not, why bring it here? If it was, then what went wrong in transit?

One hopes that the need to ask these and many other questions is due to nothing worse than ignorance, and that the answers are available. Nevertheless, it is hard to recall a really impressive advance attributable to the industry's own efforts.

Even the big contractors, who produced practically every worthwhile plant in the past, seem to confine their advertising nowadays to competitive lists of plant manufactured under licence from other countries.

I am still puzzled by what the report describes as a difficulty in the way of a national grid. Surely, even without access to refinery gas and the rest, no two area boards would arrive at precisely the same cost per therm fed to the grid.

There can always exist, therefore, the case that a lower price board will at times have to accept gas from the grid at an uneconomical rate by its standards—provided that it sells to the grid at its own cost into main.

The alternative seems to rest with the separate grid authority which has been suggested. By fixing an average price for all suppliers it can offer not a difficulty but a real blessing, in the incentive to the higher price boards to find ways of cutting manufacturing costs.

Gone too far?

Perhaps the industry has gone too far in giving the separate boards autonomy in attempting to balance their books. The comparative figures are splashed around in the daily papers at regular intervals, together with brief apologetic excuses for losses and modest acknowledgments for profits.

It so resembles a rather gentlemanly football cup competition that one tends to lose sight of the main object, which is the net overall result. Coal and electricity probably have their local inquests, but seem to be more discreet about them.

The only purpose and justification for a grid, and, indeed, for the gas industry itself, is to provide for the nation a fuel service as cheaply and efficiently as it can.

The nation has a right to expect that parochial considerations and differences shall not stand in the way of

this objective. If there are to be difficulties, they must be removed firmly by internal action, not passed on to the consumer.

One more remark in the survey calls for comment, whether it be taken as a half truth or as a fatalistic acceptance of something which should not be accepted.

It reads: 'Other costs—distribution, consumer service, administration and so on—have risen and are rising, but are not directly related to the quantity of gas sold.'

On the matter of administration, Professor Parkinson has written with sufficient eloquence for it to be passed over here. But distribution and consumer service are not so well served.

Not many years ago it was discovered that both these factors were quite closely related to therms sold, and the discovery led to a mild revolt against domestic consumers, and the wooing of industry.

It is only in very recent times that the gas industry has awakened to the fact that it is being cold-shouldered in turn by the domestic market, and is now setting out to win back, or at least retain, this better than marginal business.

This is the way to restore the *status quo*, to relate costs to therms sold, and it calls for new thinking. The inclusion of consumer service as a rising cost is surprising, for one has the impression that one pays for all this nowadays. I should object to paying for the clearance of a water-logged service or other natural catastrophe, but with modern manufacturing plant should not expect such things to occur.

We must take it that something occurs beyond the meter which is a charge upon the industry, and is of sufficient statistical consequence to warrant inclusion under 'other costs.' One has, reluctantly, to refer again to coal and electricity, because of their externally similar structure, in order to point out that they seem to have avoided any remarkable debit in a like context.

Their difference is that they know when it pays to end their responsibility: The coal industry at railhead, electricity at the meter. The gas industry is so busily engaged in throwing overboard traditions and accepted practices that it can hardly refuse on logical grounds to re-examine its position in relation to this also.

There is, of course, a special skill required to get the best out of a gas appliance. But if it is any more special than the skill required to repair a fault in a radio set, then I shall lose confidence myself and join the defaulting domestic consumers.

Taken as a whole, the symposium of reports presents a wide and properly optimistic picture of the gas industry. We all believe in gas, and in the future of gas, and one cannot fail to be impressed by the way in which strategic planning is leaping ahead.

While the sights are so firmly fixed on the horizon, though, there is an uneasy thought that nobody can be bothered about the less glamorous job of tidying up.

REVISED BRITISH STANDARD

Domestic space heating by means of independent gas appliances

ALL people concerned with the use of town gas as a heating medium will be particularly interested to hear that the British Standards Institution has just issued a new edition of the Code of Practice for domestic space heating by means of independent gas appliances (C.P. 332.301).

It must be remembered that this code does not deal with central heating by gas, which is the subject of a separate code, C.P. 332.303, or with warm air systems.

The code takes into account advances made in the design and range of space heating appliances and developments in installation technique since its initial publication in 1947, and stresses the importance of consulting the gas undertaking at the early planning stage. The whole of the code recommendations are framed to meet the thermal requirements of the Model By-laws.

Throughout the code three main methods are discussed. These are:

1. **FULL HEATING,** which is defined as a single appliance capable alone of providing the necessary standard of heating at all times.
2. **BACKGROUND HEATING** by means of an appliance or system used to provide a level of warmth lower than that needed for full thermal comfort, or supplementary heating meaning an appliance used to provide additional heating to that already present as background heating so that full comfort can be achieved when required.
3. **SELECTIVE HEATING** refers to the siting of suitable appliances so that heat, normally in the form of warm air, can be directed into more than one room or locality.

In the design section, information is given on desirable heating requirements to achieve comfort conditions in various rooms of domestic premises according to the degree of occupancy.

The types of appliances capable of providing these heat requirements are detailed, and ample supporting tables are provided for calculation purposes. These appliances include radiant and convector heaters of both the hearth and panel type, room-sealed heaters, air heaters, balanced-flue heaters and portable appliances.

Information is given on fixing both the flueless type heaters and those to be connected to a flue.

The code also carries an appendix where the methods of calculating heat requirements are fully explained, as also are such matters as fitting gas fires to existing brick chimneys and the use of restrictors; precast concrete flue blocks for hearth or panel fires, and conventional flue systems for very tall blocks of flats.

The code concludes with a series of diagrams illustrating fixing details for hearth and panel fires and the use of ridge terminals in roofs.

Copies of this standard may be obtained from the British Standards Institution, Sales Branch, 2, Park Street, London, W.1, price 10s. each. (Postage will be charged extra to non-subscribers.)

From a paper presented at the I.G.E. Autumn Research Meeting

Modern analytical techniques applied to the gas industry

PT. 1—ANALYSIS OF P.F.D. BY GAS CHROMATOGRAPHY

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THE use of p.f.d. as a feedstock for gasmaking in carburetted water gas and Segas plants has now become widespread owing to the rise in price of gas oil and heavy fuel oil and the increased demands by other industries on these oils.

As the specification to which p.f.d. is supplied from the oil companies is very wide, different batches of p.f.d. could vary considerably in composition. It is important, therefore, that a complete analysis of a p.f.d. should be available so that gas-making conditions can be adjusted accordingly.

Chromatography is at present the only technique that can give this kind of comprehensive analysis, and it is already of great importance in research in the gas industry for rapid analysis of gas, feedstocks, benzoles and tar fractions.

Key part of apparatus

As gas chromatography is essentially a method of separation, the key part of the apparatus is the column on which the separation is effected. The rest of the apparatus (*i.e.*, detector, amplifier and recorder) are merely for monitoring and recording the components as they are eluted (*i.e.*, as they emerge) from the column.

The column consists of a graded inert support, in this case 60 to 80-mesh brick dust, on which is supported a small amount (usually about 10%) of an organic liquid of low vapour pressure called the 'stationary phase,' the choice of which depends on the analysis required. The components of the mixture to be analysed are separated by virtue of the differential partitioning effect between the carrier-gas phase and the organic-liquid stationary phase.

In this case, it was known that the p.f.d. consisted mainly of saturated paraffins with, possibly, some aromatic components. No olefinic compounds were believed present, nor, in fact, were any found.

The two stationary phases chosen were (*a*) Squalane (a C_{30} paraffin) a non-polar compound and (*b*) Polyoxyethyleneglycol-400-Dibenzoate, a strongly polar substance.

The columns were 4 ft. by 4 mm. and of glass. The stationary phase used amounted to 5% in the case of squalane, and 10% for the glycol. Efficiencies of approximately 2,000 theoretical plates were obtained.

The detector used was an argon ionisation detector, designed on a principle developed by J. E. Lovelock[1].

The carrier gas, argon, passes through a small cell in which is mounted a source of ionising radiation—strontium 90. Ionised as well as metastable excited argon atoms are produced in the cell. The ionisation potential of most organic molecules is less than the excitation potential—11.6 eV—of argon. The presence of traces of organic gases or vapours in the cell thus results in ionisation of these gases by transfer of energy from the excited metastable argon atoms.

If a collecting electrode be placed inside the cell and a voltage applied across it, a small standing current will flow when argon alone is passing through, owing to the small concentration of ionised argon atoms. When an organic gas is eluted from the column and enters the detector cell, the result is that the standing current is increased and after amplification, it gives a 'peak' on a potentiometric recorder. The area of this peak is proportional to the weight of vapour entering the cell. In this way, each component is detected and recorded as it emerges from the column.

The two problems in chromatography that have to be solved before an analysis can be carried out, assuming a suitable column has been prepared, are identification and quantitative estimation of the peaks obtained for the mixture.

For a given column, flow rate, temperature and pressure, a given substance will emerge from the column in a definite time, this time being called the 'retention time.'

Retention volume

The typical constant of any substance is its 'retention volume,' that is, the volume of gas that has passed through the column from the start of the analysis to the time when the peak emerges from the column.

Identification can be carried out by obtaining these 'retention volumes' by chromatography of the pure compounds under standard conditions of column preparation, flow rate, temperature, pressure and chart-speed. These are then compared with those calculated from the mixture peaks under the same standard conditions.

In practice, it is easier and quicker to compare the retention volumes of the pure substances with that of a standard, thus calculating a relative retention volume (Rt_B).

For an homologous series of compounds, graphs of log R_{IB} against boiling-point generally give straight lines. Knowledge of an unknown peak's relative retention volume can, therefore, give a fair estimate of its boiling-point and thus enables it to be identified. This is assuming that a good guess has been made as to its nature, *i.e.*, whether it is a paraffin, *cyclo*-paraffin, olefine or aromatic.

With p.f.d., the possibilities were not so wide, as no olefines and only perhaps benzene and toluene of the aromatics were present.

Perhaps the best way of identification is the infra-red method. Each peak as it is eluted is collected after the detector in a cold trap and subjected to infra-red spectroscopy for identification. Unfortunately, the very small sample sizes used for this detector precludes, at the moment, the use of this technique.

Paraffins identified

Using the retention data obtained for the analysis of p.f.d., it was possible to identify individual paraffins up to *n*-Heptane, after which the number of isomers became too great to obtain individual peaks on a 4-ft. column. Usually, however, the peaks for the straight-chain paraffins were definable. The mass of peaks preceding each of these paraffins was assumed to correspond to branched-chain isomers of the same carbon number.

The response of the detector is such that theoretically the mass of substance entering the cell is proportional to the resultant peak area. The response should be similar for members of a homologous series and, in fact, similar for various types of compounds; that is, a mixture composed of equal parts by weight of *n*-pentane, *cyclo*-hexane and benzene should give a chromatogram that has three separate peaks, all of which have the same area. If this is not so, the compounds have different responses and individual calibration is needed.

To determine to what extent this was true, and to find out if individual calibration was necessary, a series of standard mixtures was made up and chromatographed. The peak areas were measured by the product of height and half width. The sum of the areas was found and each peak area was expressed as a percentage of the total. This should, theoretically, give the correct analysis figures as *w*/*w* percentage, a method known as the 'internal normalisation.'

Two varieties of p.f.d. have been used for carburetted water gas and Segas feedstocks in the Leeds area; they are both low in aromatics and have sp. gr. of .68 and .72 respectively. Different varieties have been described by other workers.[2]

Up to about September, 1960, the p.f.d. with a specific gravity of about .68 was used in Leeds, after September, 1960, the source of supply was changed and the p.f.d. supplied now had a specific gravity of approximately .72.

A comparison of the two analyses shows that the oil companies had widened the earlier p.f.d. range to include some of the petrol fractions. When the .72-sp. gr. p.f.d. was first used in Leeds, some trouble was experienced with condensation of a heavy oil in carburetted water gas separators. On analysis, this oil was shown to represent the difference between the two p.f.d., and the heavy end of the .72-sp. gr. p.f.d. were obviously coming through uncracked. Adjustment to the cracking conditions was then made, and no more condensate resulted. This illustrates just one of the applications of comprehensive feedstock analysis as described in this paper.

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From a paper presented at the I.G.E. Autumn Research Meeting.

Analysis of oils for gas manufacture

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THERE are two ways of expressing the hydrocarbon-type analysis of an oil if there are mixed-type molecules present. These may be called hydrocarbon-type and carbon-type classifications. For instance, by the first classification, hexylbenzene and tetralin are both 100% aromatic, because each molecule contains an aromatic ring. By the second classification, they are 50% aromatic/50% paraffinic and 60% aromatic/40% naphthenic respectively. The second classification would appear to be more appropriate from the gas-making point of view.

The hydrocarbon-type classification may be further subdivided into volume percentage, weight percentage and mole percentage. The carbon-type classification is essentially weight of carbon percentage.

The unsaturated content is normally very low, and it is not known whether the unsaturated hydrocarbons are mainly ring structures or open-chain. They have, therefore, been neglected in this discussion.

Ring-type sulphur compounds are considered as aromatic or naphthenic according to structure; thioparaffins and mercaptans are considered as paraffins. For gas oils, the molecular weight is about 210, so that the normal sulphur content of .8% corresponds to 5% of sulphur compounds w/w.

Gas oils are such complex mixtures that any attempt to determine all the individual components would be impracticable. Analysis must, therefore, involve the separation of the hydrocarbons into types, or the use of physical correlations that have been established with pure components or pure types.

The Griffith method of analysis is the only method that has been proved to give good correlation with enrichment

values over a wide range of oil composition. The introduction of a specification based on the method made a great improvement in plant performance, and the fact that it gives comparative data for composition is much more significant than its absolute accuracy.

The analysis is done on a 95% distillate, and involves removal of the unsaturateds with 80% sulphuric acid, solution of the aromatics in activated sulphuric acid and determination of the naphthenes in the residues by comparison of the aniline-point with those for supposedly pure paraffins and naphthenes of the same mid-boiling point.

The determination of the naphthenes in the saturated residue from the aniline-point depends on the use of calibration curves obtained with pure paraffins and naphthenes.

Further evidence that the naphthene contents are too low is given by the ring numbers R_N (average number of rings per molecule) of the saturated residues after acid washing, calculated from the percentage hydrogen (H) and the molecular weight (M).

Possible alternatives

Supposing that the aromatics are determined by the Griffith method, there are various possible alternatives for determining the naphthenes and paraffins, either by measurements on the saturated residue from the acid wash or on the original oil.

Such means are (*a*) Revised calibration curves. (*b*) The use of characterisation factors K—which is a measure of the composition of an oil which can be read off from nomograms using various pairs of physical properties, such as specific gravity and viscosity, mean molal boiling-point, molecular weight or calorific value. (*c*) The use of viscosity/gravity constant (VGC) which is again a simple method, but appears to give rather less consistent results than the use of K. (*d*) The aniline-point and molecular weight of saturated residue. (*e*) The use of the refractive index, density and molecular weight of the saturated residue, a method which is essentially the same as (*d*). (*f*) The measurement of the refractive index and density of the saturated residue. (*g*) The molecular weight and the percentage of hydrogen in the saturated residue.

In the fluorescent indicator absorption (FIA) method the lengths of the zones occupied by the paraffins plus naphthenes, unsaturateds, and aromatics in a silical gel chromatographic column are measured directly. Fluorescent marker dyes that travel with the different hydrocarbon types are added so that the zone boundaries can be observed.

Thus, the method gives the percentages of aromatics and unsaturateds in the oil, but does not distinguish between paraffins and naphthenes. It does not yield a saturated residue for further examination by other methods, but the percentage of aromatics can be combined with measurements of K or VGC for the original oil.

For gas oils, the FIA method does not give very sharp zone boundaries and appears to have little to recommend it as an alternative to the Griffith sulphuric acid washing method for determining aromatics. It does, however, give much the same result.

The direct method of Van Nes and Van Westen involves accurate determination of the percentage of hydrogen and molecular weight before and after catalytic reduction of the aromatics to naphthenes. This reduction must be carried out slowly in stages to avoid breakdown of the naphthenes. Since each aromatic C atom will take up one H atom on reduction, it can be shown, without any further assumptions, that the percentage of aromatics $= 1201\ (-H+M'H'/M)/1.008\ (100-H)$, where H, H', M, M' are the percentages of H and molecular weights before and after reduction. A correction can be made for sulphur content.

The method is much too complicated for general use, but forms the basis for a number of other methods. Oils analysed by the direct method, have been used as standards in developing these other methods.

A number of papers have been published on the infra-red analysis of oils, making use of the different absorption bands of aromatic and paraffinic groupings. Unfortunately, in the present state of infra-red spectroscopy, it is not possible to use extinction coefficients obtained by other workers with different instruments, so that a calibration is required by each worker, using hydrocarbons of known composition in the correct boiling-range.

The authors have, however, made some measurements by the method of Brandes, using the absorption bands at 720 cm^{-1} to determine the paraffins and at 1,610 cm^{-1} to determine the aromatics. The naphthenes were determined by difference.

This method requires special equipment and pure-type oils or analysed samples for calibration, but it is very quick.

Approximate methods for carrying out these analyses include (*a*) The characterisation factor (K) from which a rough value for P—the percentage of paraffins—can be found. (*b*) The viscosity gravity constant (VGC) and (*c*) the C:H ratio, by which an equation is derived for the C:H ratio in terms of the composition, but it is necessary to make assumptions about the type of ring structure.

The original purpose of this work was to devise a simple procedure for determination of the paraffin content of gas oil, which would be satisfactory for routine control purposes. It was necessary that it should give the same results as the Griffith method (now believed to be too high), so as to fit in with an existing specification.

This has been achieved by use of the approximate viscosity gravity constant (VGC) method with suitable adjustment of the constants. The percentage of paraffins in the oil is taken as 283 (2.097—VGC), but, to avoid the impression of bogus precision, this is called the ' valuation number ' rather than the percentage of paraffins.

The method has been in use for about five years by the North Thames Gas Board, and has been found to give consistently good agreement (within 1 or 2 units) with occasional checks by the Griffith method.

The procedure is to measure the specific gravity of the oil at room temperature, accurate to .001 units, by means of a hydrometer; the reading is then corrected to 60° F. in the normal way.

Kinematic viscosity

The kinematic viscosity in centistokes is then measured at 100 ± 1° F. The viscosity-gravity constant and the valuation number can then be read off from a nomogram.

The *n-d-v* method of Cornelissen and Waterman is recommended for more precise analysis. This involves accurate measurements of refractive index (n) and density (d), both at 20.0° C., correct to .0002 units, and of kinematic viscosity (v) in centistokes at 20.0° C., correct to .5%. It uses the same approach as the *n-d-M* method of Van Nes and Van Westen, but it is very much easier to measure the viscosity than the molecular weight (M).

The refractive index can be measured with a water-jacketed Abbé refractometer with water at 20.0° C. streaming through the instrument. The density is conveniently measured with a 5 ml. graduated pyknometer, suspended in a Thermos flask of water at 20.0° C.; the instrument is previously calibrated with mercury. The viscosity can be

measured with a National Physical Laboratory-calibrated suspended level viscometer.

Methods of hydrocarbons-group analysis suitable for gas oils cannot be applied directly to light distillates, owing to the great difference in molecular weight of the component hydrocarbons. The authors first used a method based on that of Kurtz, which required preliminary fractionation into eight cuts. However, apparently more satisfactory results were obtained by the method of Lipkin, Martin and Kurtz from the temperature coefficient of the density and either the density or the refractive index. The temperature coefficient of the density was deduced from the molecular weight, which in turn was deduced from the density and mid-boiling-point.

These methods of analysing the saturated residue were confusing and indirect, and there was little to indicate if the results were reliable. In 1957, the authors consulted Mr. H. C. Rampton, of the British Petroleum Company, Ltd., and eventually adopted a method based on his 1949 paper.

Residue is analysed

It is first desirable to remove the very low-boiling components so as to be able to make measurements on the residue without loss by evaporation. The volumes of residue and distillate are measured, any small loss being included in the latter. Only the residue is then analysed further, it being assumed that the distillate, in view of its boiling-range, can contain no naphthenes or aromatics. The unsaturated hydrocarbon content of the distillate can be determined by means of the bromine number, but is normally too low to be worth considering.

The percentages by volume of unsaturateds (normally small) and aromatics in the distillation residue are then determined by the FIA method; quite sharp boundaries are obtained. Alternatively, the percentage or aromatics plus unsaturateds may be determined by washing with activated sulphuric acid. Both methods give similar results.

The remainder of the distillation residue is then de-aromatised by passage through about 50 cc. of 100- to 200-mesh silica gel, as used in the FIA test.

The first 15 ml. of eluent are collected separately, and are used for accurate measurement of density (d) and refractive index (n), correct to ± 0.0002 units, at 20.0° C.

The remainder of the de-aromatised distillation residue is used for determination of mid-boiling-point in a simple distillation. Having completed the analysis, it is necessary to work backwards to the original oil.

This method would appear to be sound in principle, and the results obtained over a period of three years agreed well with the calculated characterisation factors K. It is apparent that the method is unsatisfactory for low-boiling materials—final boiling point 60° C. In any case, they contain relatively few components, so that they can be analysed successfully, and in much greater detail, by gas—liquid chromatography.

As in the case of gas oil, the authors were asked to devise a simple method of analysis that would give an approximate value of the paraffin content satisfactory for routine control purposes.

Investigation showed that the viscosity/gravity constant was unsuitable for this purpose. Moreover, it is difficult to measure the viscosity of low-boiling oils at 100° F. because vapour bubbles may be formed in the capillary of the viscometer.

Use was therefore made of the characterisation factor, K, which is a measure of the composition of an oil. It was originally defined as the cube root of the mean molal boiling-point in degrees Rankine (°F.+460) divided by the specific gravity at 60° F.

The expression for K in terms of the hydrocarbon-type composition varies with boiling-point, so that we cannot use the same expression as for gas oil. For hexanes to nonanes, normal or with one methyl side chains, $K=12.7$. For mono-and di-substituted *cyclo*pentanes and *cyclo*hexanes $K=11.4$. For toluene and xylenes, K may be taken as 10.2. Thus for a mixture of hydrocarbons, such as light distillate with a mid-boiling-point of about 100°C,

$$K=0.127P+0.114N+0.102A,$$

where P, N and A are the percentage contents of paraffins, naphthenes and aromatics respectively.

Again, to avoid an impression of bogus precision, this is called the valuation number rather than the paraffin content.

The sulphur content of light distillate is critical in connection with new methods of continuous reforming and it is desirable to be able to measure down to .1 ppm. To do this, it is necessary to burn a large sample and also to have a sensitive method of analysis.

The Wickbold burner provides a means by which a 20 g. sample of light distillate can easily be burnt in about 30 minutes; this is very much faster than by the lamp method.

The sulphur in the sample is burnt to sulphur oxides and absorbed by hydrogen peroxide solution in an absorber.

Various methods can be used for determining the sulphuric acid formed in the absorber. Wickbold describes a sensitive nephelometric titration in which dilute barium chloride is titrated in until there is no further increase in turbidity. This is much more reliable than an absolute measurement of turbidity which is very sensitive to the operating conditions. The authors have modified the method slightly to increase the sensitivity.

The accuracy of the method depends on the completeness of absorption, loss in handling, titration error and blank. A definite titration error was observed with a measured volume of .002N sulphuric acid; the results were about 4% low. The overall error was determined with a standard solution of thionaphthene in sulphur-free benzene, equivalent to 6 ppm. of sulphur; the results were 6% low.

The sensitivity of the method is controlled by the size of the blank. The end-point can be determined to .02 ml, which corresponds to .04 ppm. of sulphur on a 20-g. sample. However, the blank, determined by burning sulphur-free methanol, may be as high as .1 ml., corresponding to .2 ppm. of sulphur on a 20-g. sample. It should be possible to determine whether the sulphur content is more or less than .1 ppm. by burning a 50-g. sample.

DISCUSSION

Mr. G. R. Boreman, London Research Station, considered that after six years' experience of gas chromatography it was simple to obtain qualitative interpretations from a chromatogram and fairly simple to obtain semi-quantitative data—± 10%—but it was not easy to obtain truly quantitative data, particularly on complex mixtures, such as p.f.d. or crude benzoles.

To obtain accurate results, even for simple mixtures, detector response had to be linear for each component over a wide range of concentration and relative sensitivity—the difference in signal strength obtained for the same weight of each compound—had to be accounted for.

Referring to the work of other investigators he said that these sensitivities might vary for even simple C_7 and C_8 paraffins by as much as 20%.

It would have been helpful to have had a chromatogram included in the paper, which would have given more indication of separation and time taken for analysis, and to have known to what hydrocarbon the 2,000 theoretical plates referred, and he was quite sure that 4-ft. columns were not long enough to give p.f.d. separation without danger of considerable overlap in the higher carbon ranges. There were 35 possible C_9 paraffins, and in his own laboratory 29 had been counted as present in one p.f.d. fraction separated on a 300-ft. squalane-coated capillary column. Thus [on p. 6] some components of the 16% C_9 paraffins quoted could quite easily interfere with the *n*-octane and *n*-nonane peaks. More important still, most p.f.d. contained not only paraffins but also olefinic unsaturated components, naphthenes and aromatics which would most certainly interfere in an analysis of this kind.

The complexity of these oils, both in range of carbon number and in chemical type, made it necessary to use longer columns than those quoted, and to use a detector which had been carefully calibrated.

His own choice for this analysis would be the simple hydrogen flame detector, which afforded two immediate advantages: (a) The relative response towards hydrocarbon on different chemical type was much closer than the argon detector and its response over wide ranges of component concentration was more linear, and (b) a larger sample was required—about 5-10 mg.—which added to the inherent accuracy of the analysis.

Mr. T. A. Dick, West Midlands Board, referring to the classic Griffith analysis of gas oils which had proved of such great value in assessing their suitability for enrichment of water gas, said that there was a more fundamental reason why its shortcomings did not affect its usefulness and suggested that this was because the naphthenes contributed by dehydrogenation reactions at least as much as the olefine to the production of gaseous therms under the enriching conditions of the c.w.g. plant. Probably a more useful guide to the unsuitability of any oil for town gas manufacture was its content of aromatics.

Aromatics did not readily break down or hydrogenate —modern hydrorefining of benzoles was a classic example of this. Where the proportion of non-aromatics in the oil was high the maximum thermal yield turned less on hydrocarbon type analysis than operating the plant in the most efficient manner possible.

If this explanation was correct, the significance of the authors' many empirical derivations of the hydrocarbon type content of oils became less apparent, and it was very difficult indeed to draw conclusions as to the best valuation number to be placed on the six gas oils detailed [in Table 2]. The distinction between the various types of paraffins, aromatics and naphthenes must in any case fade as the molecular weight of the petroleum distillate or residue increased.

The authors' confirmation by spectrophotometric means that the aromatics in gas oil were completely removed during the acid washing stages of the Griffith method was interesting since the Institute of Petroleum, together with the A.S.T.M. had established the precision and limits for several methods of determining hydrocarbon types of oils.

Olefines were determined by bromination methods, aromatics by acid-washing and saturates, olefines and aromatics by the empirical fluorescent indicator adsorption method, an excellent method for distillates. For re-distilled gas oils, using the present improved technique, the results were a sufficiently good guide as to the suitability of oils for gas manufacture, but naphthenes and paraffins needed differentiating, then the authors' method using K would suffice.

There was a lack of published information on the relationship between the hydrocarbon type analysis of oils and the maximum thermal yield obtainable on full-scale c.w.g. or other oil using machines. To carry out such work, not only had optimum plant performance to be maintained throughout the tests, but large-scale supplies of like materials, e.g., light petroleum distillates differing in hydrocarbon type analysis, must also be available.

Mr. J. H. Towler, North Eastern Board, referring to the application of another modern physical method to the analysis of fuel gases, *viz.* infra-red spectroscopy, stressed that the time taken to carry out an analysis of this kind had been some four to six weeks. Using gas chromatography the time needed for a similar analysis might only be two or three days and the sample required would be greatly reduced.

The infra-red equipment used in the early work upon gas composition had cost around £3,000, but now it was possible to equip a laboratory with a gas chromatography unit for around £300—little more than the price of a potentiometric recorder.

These points should be underlined because the technique, although modern, was within the scope of any gasworks laboratory of reasonable size.

In the application of the technique illustrated in the paper the analysis of two samples of p.f.d. was a good one. Infra-red spectroscopy had been found incapable of distinguishing between complex mixtures of paraffin hydrocarbons without previous fractional distillation. The present technique did not involve fractionation and gave better results.

Dr. D. McNeil, Director, Coal Tar Research Association, while recognising gas chromatography as the greatest advance in analytical technique since the development of micro analysis, said that its outstanding successes had been as a method for separation and identification of complex mixtures, but only to a limited extent as a quantitative method for the analysis of relatively simple and relatively volatile mixtures. A panel recently set up by the Standardisation of Power Products Tests Committee under the chairmanship of Dr. Scott, the leading authority on G.L.C. in this country, was to examine critically the reasons for discrepancies in quantitative analysis. Their findings to date were rather illuminating, although rather alarming.

The internal normalisation method contained another possible source of error, since it made the assumption that all the sample injected left the column, that nothing but the sample injected left the column, and that the sample was completely separated. Such assumptions were probably reasonably sound in the analysis described by Professor Roberts, but they were not valid for many other analyses.

The analysis of p.f.d. was exactly the type for which chromatography could be extremely useful at its present stage of development. It would, however, have been much less useful to Mr. Densham and Mr. Beale, because they would not have had anything like the number of reference compounds available; nor would it have been easy to select a stationary phase capable of giving the necessary separation without it either decomposing or volatilising under the higher temperature conditions.

Mr. H. C. Rampton, Assistant Section Head, Analytical Section, B.P. Research Centre, referring to the paper on the determination of the composition and character, and the estimate of the value of oils for gas manufacture, gave some idea of the complexity of the problem when he spoke of the possibility of there being just over 10,000 isomers of

hexodecane, $C_{16}H_{34}$, alone in the narrow boiling range gas oil. To this should be added the number of the remaining paraffins, the naphthenes (5- and 6-membered ring, monocyclic and polycyclic), the aromatics (monocyclic and dicyclic) and mixed types; the total figure of components became astronomical.

Gas chromatography was not the analyst's alchemist's stone that people imagined, and he defied anyone to identify possibly 1,000 peaks from some petroleum oil by gas chromatography alone. Some other technique was needed.

Even if a completely detailed analysis was obtainable, the final step had still to be the reduction of these figures to some form of gas-making index. The best line of attack therefore was a compromise, in the form of a structural group or carbon type analysis, akin to the ring analysis proposed by other investigators.

The FIA method was not satisfactory for oils containing appreciable quantities of materials boiling over 275° C., because of the empirical nature of the dyes employed, which were prepared to be of roughly the same general absorbability as the aromatics. The determination of molecular weight accurately was both tedious and demanded a high level of operator technique; the carbon/hydrogen ratio would be satisfactory, but the accuracy necessary was virtually impossible to achieve.

He asked the authors whether the precision of their method for the determination of low sulphur contents had been established and if so, whether it had been appraised statistically from results obtained (*a*) by highly skilled analysts using extreme care and a few samples; or (*b*) by routine operators on a large number of samples.

Miss N. H. Farrow, South Eastern Board, referring to the work being done in the central laboratories of her Board on the analysis of primary flash distillate feedstock by gas liquid chromatography, said that they had determined the composition of feedstocks by correlating analyses using a column of non-polar stationary phase for paraffin selectivity with those using a column of polar stationary phase for aromatic selectivity; they had obtained substantially similar results.

This technique could be applied to a particular gasworks problem, namely, the study of the performance of carburetted water gas plants operating on these feedstocks.

By obtaining the analysis of a feedstock and that of a cryoscopic condensate of the actual gas made from this feedstock in a carburetted water gas plant, they had been able, provided the amount of condensate per cu.ft. of gas was known, to obtain very valuable information on the completeness of decomposition of various hydrocarbon types in the plant.

The general conclusion had been that about 95% of the feedstock could be decomposed when making gas of calorific value 500 to 550 B.t.u. per cu.ft., but that there had tended to be a noticeable decline when approaching a calorific value of 600. The evidence had indicated that different types of hydrocarbons contained in the feedstock did not differ significantly with regard to their ease of decomposition, though the benzene content of the gas had represented rather more than had been present in the feedstock, thus indicating that additional benzene had been formed in the plant.

Dr. R. G. Cockerham, Organic Chemist, Midlands Research Station, said that the retention ratios obtained by the authors agreed well with those obtained by other investigators using similar operating conditions, but he did not altogether agree with their proposals for quantitative measurements, although he did accept that with an argon ionisation detector, the method of internal normalisation did give the composition by weight, without great error.

Since the detector response varied with molecular weight and ionisation cross-section of an individual hydrocarbon, the method should be confined to mixtures of components of about the same molecular weight. Variations produced by the two factors were then small, and in any case were in opposite senses, and the detector response was almost linear with weight of hydrocarbon applied to the column.

He asked the authors whether they were justified in applying the internal normalisation method to a mixture containing components which had as wide a range of molecular weight as their higher gravity p.f.d. and the heavy oil condensate. If as revealed by analyses the heavy oil condensate comprised almost exactly half the p.f.d. he thought that the engineer concerned must have had for a time a remarkably inefficient enrichment stage in his c.w.g. plant!

He asked Mr. Densham why he had abandoned the parameter refractivity intercept in favour of specific refraction, when calculating the percentages of naphthenes and paraffins in a dearomatised fraction. It was a simpler expression, and led to no greater error. To minimise error the refractive index should be measured as accurately as possible, so it was significant that in a similar method published by Universal Oil Products Ltd. refractive index was to be measured to five places of decimals.

He could confirm that the method published for sulphur did indeed work, in fact it worked extremely well, but they were concerned with the catalytic gasification of a very pure distillate which was purified specifically for use with a catalyst extremely sensitive to sulphur.

Though the title of their paper was all-embracing, Mr. Densham and his co-author had confined their field to hydrocarbon type analysis, and had not mentioned the large number of other properties of interest to engineers which might be deduced from their valuation number K, and one or two simple measurements. Such properties included specific and latent heats, and critical properties.

Mr. K. F. Buttall, North Thames Board, said that at Beckton they too had been engaged on the analysis of p.f.d. by gas chromatography. The apparatus utilised the hydrogen flame thermocouple detector in conjunction with a 50-ft. column. The stationary phase and solid support were 17% Apiezon L on 60/72 mesh Celite and the column temperature was maintained at 77° C. Some 33 components had been detected from C_4 to C_8 of which 16 have been identified to date.

A light distillate, of mid-boiling point 54° C., recently analysed with this apparatus, had been found to contain 12% naphthenes and only 1% aromatics. These agreed with the figures quoted by Mr. Densham [in Section IV], in that the naphthene content of these lighter distillates was in actual fact higher than shown by other methods of analysis and therefore it did not seem to be strictly correct to use the approximation that $N=A$ to arrive at the equation $P=40\ (K-10.4)$ for the valuation number.

Referring [to Section VI] a Wickbold apparatus had been in use at Beckton for some time for the analysis of p.f.d. and for butane.

The method of determining the sulphuric acid formed in the absorber involved the titration of the sulphate with lead nitrate solution using dithizone as an indicator.

First the absorber solutions and washings had had to be evaporated to dryness to destroy the residual hydrogen peroxide, and the residue was then taken up to 10 ml. of water and the method of Archer (*Analyst* 1957, **82,** 208) followed. The overall error had been determined by burning standard solutions containing 10 ppm. of sulphur.

The results had been 5% low—of the same order as Mr.

Densham's barium chloride method, but it was not as sensitive; it should be possible, however, to record 3 ppm. with a 50-g. sample. Butane normally contains about 10 ppm. and p.f.d. 600 ppm.

Mr. C. H. Lewis, North Thames Board, said that the Griffith method of analysis was now shown to be in error and their gas oil was not of such high quality as they had thought. However, it was probably the best available at the price the gas industry could afford to pay, and the main purpose of the analysis—as between buyer and seller—was to maintain the standard of quality.

The VGC method had proved of considerable advantage for routine control purposes, but it was well to remember that it had been adjusted to give results which were in agreement with the Griffith method, so that the latter had to remain the standard of comparison.

When primary flash distillate was used for carburetting without reforming, the yield of gas appeared to approach 100% of the thermal value of the feedstock. This suggested that with this material the hydrocarbon type was less important than the maximum boiling point—presumably because of the volatility of the cyclic components of p.f.d.

In the absence of reforming, the yield of gas from p.f.d. could presumably be evaluated by the Pacific Coast method —B.S. Test Code No. 1784—and possibly in some of the area boards there was sufficient data to enable the gas-making results to be correlated with the quality of the p.f.d. used. If so, the early publication of such results would be of value to these responsible for the operation of water-gas plants.

In catalytic gasification plants, the relation between feedstock composition and plant behaviour might be quite complex, and both choice of process and economic operation might depend on their knowledge of feedstock quality. Studies such as these now published by the authors of these two papers was therefore of more than academic interest.

Mr. A. G. D. Yeaman, South Western Board, said that they employed several gas liquid chromatographic columns with lengths ranging from 10 ft. upwards, each operating at a different temperature to enable them to analyse a wide range of hydrocarbons from liquid petroleum gases to a light gas oil.

The authors—Professor Roberts and Mr. Ward—reported [in Section V] results for two samples of p.f.d. quoting up to 24 different substances. With their routine apparatus they had distinguished up to 37 constituents with appreciably more aromatics than mentioned in this paper. But they knew that there would be 60 or more constituents in p.f.d.

In view of their success with the investigations on water-gas plants, they had carried out an extensive survey of the various hydrocarbon condensates found in the gas supplied on the district and gas produced from several other processes involving cracking processes.

So far they had had to freeze hydrocarbons out of the gas stream and then analyse a drop of the liquid obtained. In future they expected to be able to analyse the hydrocarbons direct from a gas sample without requiring the intermediate liquid stage.

Dutch install central gas distributing plant for efficient control

By J. Grindrod

TO ease the task of distributing the quantities of gas available at their plants, the Koninklijke Nederlandsche Hoogovens en Staalfabrieken N.V. (Royal Dutch Blast Furnace and Steelworks), Ijmuiden, Holland, has installed a central gas distributing station for all kinds of gas.

One of the advantages gained by the new system, which incorporates an efficient and clear system of control, is to make it easier to ensure that the various gases are burnt at places where an optimum return is obtained.

Another advantage is that, in the event of trouble, it is possible to act rapidly, thereby increasing the reliability of the whole process.

An additional result of operating the gas distribution station has been to reduce from 0.65% to 0.12%, the amount of total blast-furnace gas, available for distribution, which has escaped uselessly from the chimneys.

In July 1960, a natural gas transmission line from Hoofddorp to the Ijmuiden works was brought into service for conveying natural gas to be supplied to Hoogoverns by the Staatsgasbedrijf (State Gas Undertaking) and which is mainly to serve the nitrogen fertiliser manufacturing plant of Mekog (an associate company), where equipment for the processing of natural gas has been brought into use.

Mainly because of an increase, begun in 1959, in the delivery of gas to the City of Amsterdam, total Hoogovens gas sales to municipalities rose in 1960 to 278 mill. cu.m., representing an increase of about 27% compared with 1959. The price of gas declined considerably.

KITCHEN DESIGN CONTEST

A 10,500-mile telephone call from Sydney, Australia, booked 30-year-old James Pratt, of Roseville, New South Wales, a place in a catering course at the South Devon Technical College, Torquay.

It has now been announced that he is among the nine award winners from the college in the Gas Council's national competition to design the perfect commercial kitchen.

James, who made the call to Mr. G. McKay the principal, after meeting a former college student who was travelling in Australia, is returning home when he has completed the National Diploma course.

Fellow student John Cowie, 20, of 101, Oliver Street, Rugby, is awarded the second prize of £30, and eight other students, including James Pratt, win £10 prizes.

FROM OVERSEAS

Another overseas award winner is 17-year-old Jeffrey Juter, who hails from South Africa. Jeffrey was also one of two students who won £25 each for their progress in the first year of a catering course at Hendon Technical College.

London has nine winners. They are from colleges at Hendon, Walthamstow, Holloway and Hampstead, and include Sister Mary Hilda from the Convent of Notre Dame, Liverpool. Sister Mary, who passed her Institutional Management examination this summer, is now working at a teachers' training college in Liverpool.

FIRST PRIZE

The first prize of £50 in the competition, which is the second to be held in connection with the Hotel and Catering Exhibitions at Olympia, goes to 20-year-old Christopher Ducat of the Scottish Hotel School, Glasgow. The third award of £20 is made to Miss Sheila Beetham from the National Society's Training College for Domestic Subjects (Berridge House), Hampstead.

A special trophy to be held for the two years' between competitions and a certificate go to the Scottish School.

There were 304 entries, and the models of the first three prize-winners will be shown on the Gas Council's Stand at next January's Hotel and Catering Exhibition. The prizes—in Premium Bonds and National Savings Certificates—are to be presented at a reception in London the same month.

THREE DIVISIONS MERGED

West Midlands Board announces major changes

MAJOR changes in the organisation of the West Midlands Gas Board have been announced by its Chairman, Mr. C. H. Leach, and will take effect from January 1. The changes follow the appointment of Mr. F. Harvey, General Manager of the Walsall Division, as Engineer of the Lurgi Plant now under construction at Coleshill, and the request of Mr. J. E. Wakeford to be relieved of the responsibility of managing the Birmingham Division, on medical grounds.

The Walsall and Birmingham Divisions, thus left without a Divisional General Manager, have been merged with Wolverhampton Division into the Board's Central Region, which in future will operate as one co-ordinated unit.

Mr. Stanley Jones, General Manager of Wolverhampton Division, has accepted an appointment as one of two Group Production Engineers in the new Region.

The Board's present Chief Engineer, Mr. H. Moys, will be responsible for the production of gas in the 15 stations in the new Central Region and will be assisted by Mr. S. K. Hawthorn, formerly Birmingham Divisional Engineer, who has been given a new appointment as Production Engineer.

CO-ORDINATION

The production stations will continue under the management of station engineers, but their activities will be co-ordinated for Mr. Hawthorn by two group production engineers.

Mr. F. Bell, the Board's Chief Distribution and Installation Engineer, will be responsible for distribution and for gasfitting, installation and maintenance work. He will operate through Mr. F. L. Atkin, Chief Distributing Engineer, and Mr. W. J. Parsons, newly appointed as Installations Engineer, and previously Birmingham Divisional Sales and Service Officer.

Mr. W. H. Elkerton, Director of Marketing, will control all appliance sales, stores and meter and appliance repair shops.

INDUSTRIAL SALES

Mr. A. C. Jennings, the Board's Industrial Gas Officer, will be responsible to him for the industrial sales organisation in the Region, and Mr. D. N. Maddocks, Area Coke Manager, for coke sales.

Mr. A. Gray, Coal and Transport Officer, will control transport throughout the Region.

Outside the new Central Region the remaining four of the seven Divisions into which the West Midlands has been divided since vesting will continue as at present, with the exception that Mr. G. Sugden, Chief Accountant, will be responsible under Mr. W. Hyde, Director of Finance and Administration, for all the Accountancy Departments throughout the Board.

NEWS SUPPLEMENT

S.G.B. NAMES YEAR'S TOP APPRENTICES

NINE young men from the five Regions of the Southern Gas Board received recognition for their hard work recently when they received awards for being the best gasfitting apprentices of the year.

They are making Board history, because they are the first to receive certificates under the Board's own gasfitting apprentice award scheme.

The scheme will give awards to the best apprentice in both the Final and Intermediate Examinations in each Region every year.

Before presenting the certificates, Mr. B. G. H. Clegg, Commercial Manager, told the apprentices that the gas industry has a great future, and that they can have a great future in it.

Mr. Clegg stressed that the Southern Gas Board has young men at top management level, who are prepared to take a chance on young people. 'If a man is good, he'll get ahead,' he said, 'and the younger the better. We want to be a young Board, and an opportunity Board.'

Apprentices who received certificates and awards were: James Tester, for the Final Examination in Dorset and Bournemouth Region; William Griffiths, for the Final Examination and Michael Winterman, Intermediate, from Southampton Region; Brian Southwell, Final, and Barry Wheeler, Intermediate, from Wight and Portsmouth Region; Barry Dickinson, Final, and Donald Scott, Intermediate, from Reading Region; and Richard Smale, Final and Desmond Langford, Intermediate, from Oxford Region.

Permit sought for smokeless coal plant

APPLICATION has been made to the Fife County Council for permission to build a carbonisation plant for the production of Rexco smokeless coal at Comrie Colliery, near Oakley, Dunfermline.

If permission is granted and current negotiations with the National Coal Board are successful, construction of the plant could begin as early as February next year and the production of Rexco early in 1963.

Rexco smokeless coal is made by the National Carbonising Co. Ltd., at plants at Thoresby and Mansfield collieries in Nottinghamshire. Supplies of the fuel in Scotland are small and a new plant at Comrie is essential if the position is to be improved.

CONTRACTORS

Main contractors for the building of the plant will be Newton Chambers & Co. Ltd., of Sheffield, the same company which built the extension to the original Mansfield plant in 1952; the main plant at Edwinstowe in 1956 and its extension in 1959.

The actual size of the new Comrie plant will depend largely on availability of coal supplies from the Coal Board. At Thoresby Colliery, Edwinstowe, Notts—the larger of the two existing plants—there are 120 workers operating on a continuous basis and the production exceeds 200,000 tons. The new Scottish plant will probably be about half that size.

BACK AGAIN

The Heating, Ventilating and Fuel Efficiency Exhibition is returning to Manchester and will be held in City Hall, Deansgate, from September 11 to 20, 1962.

AT THE SHOWS

The Scottish Gas Board Stand at the Modern Homes Exhibition, Kelvin Hall, Glasgow.

The Gas Council stand at the Dairy Show. Behind the Counter — two North Thames Board home service advisers.

SUGG TURNOVER, PROFIT RISES

TURNOVER of William Sugg & Co. Ltd. for the year ended June 31, 1961, was about 33⅓% over that of 1960, stated Mr. W. T. Mattock, former chairman and managing director of the Company. The turnover for the first five months of this year was somewhere in excess of 25% more than what it was in the same period last year.

Mr. Mattock said that when he retired last year, he felt the Company was on the border of a very large expansion, and it was indeed expanding very well.

Mr. J. Ramsay, Managing Director, said that arrangements were being made with a substantial group of companies in France and Belgium to extend the Company's markets. This would take a long time, but it would be done.

They were going to review and modernise their stores control, their costing system, and they would be taking outside help in examining their production methods.

Reorganisation was expected to start at Crawley early next year.

The profit sharing bonus last year was 13%, this year it was 14½%, and there were a lot more people in it than before.

Mr. Mattock then presented long service awards to employees who had served 40 and 30 years with the Company (see picture, left).

GAS WARMTH

Gas heating is to be installed in the newly-modernised Wellington road railway station at Dewsbury, Yorkshire.

Nife cap lamps help gas rescue squads

SINCE help is often needed in dark, confined spaces it is vital that rescue squad members have some form of lighting which will leave their hands free and not impede them. Occasionally they have to work in an explosive atmosphere when any spark or open flame must at all costs be avoided.

To solve this problem teams from the Birmingham Division of the West Midlands Gas Board are using Nife hydrogen cap lamps for practices and in actual rescue work.

Manufactured by Nife Batteries, of Redditch, Worcs., these lamps which are similar in design to a miner's cap lamp, consist of a lightweight headpiece attached by a flexible lead to the battery.

FULLY APPROVED

They are fully approved by the Factory Inspectorate for use in atmospheres containing gases of the hydrogen/pentane group (including coal gas) and are covered by Factory Department 'intrinsically safe' certificate No. IS.3003.

They are powered by Nife nickel-cadmium alkaline batteries, which retain their charge even when stored for long periods. The all-steel, robust construction of these batteries ensures many years of trouble free life.

The headpiece is normally attached to the peak of a miner's safety helmet, or, when Salvus breathing apparatus has to be worn, can be clipped on to a special headpiece retaining bracket, which can be attached to the apparatus.

LIGHT HEADPIECE

The lamp meets Home Office specification JCDD/21 covering the use of lamps with breathing apparatus. The small headpiece is very light, weighing only 5½ oz. and, on its 4 ft. 6 in. cable, can be used in the hand when searching for casualties.

Approximately 450 works operators form the fire, rescue and ambulance services of the West Midlands Gas Board. Of these, about 100 have been fully trained in their own time for rescue work and are concentrated in the larger gas-works, where they can be called away from their normal production duties at a moment's notice.

They have to attend regular weekly training drills and, so that they will maintain a constantly high standard, teams from works throughout the Board's area compete in national and local first aid competitions, achieving consistently good results.

Since no competitions exist for rescue work, the West Midlands Gas Board has initiated its own for teams from its seven divisions.

The hydrogen cap lamps are inspected once a week and it has been found advisable to charge the batteries after every four practices to ensure that they are ready for any emergency.

The lamps are used for many other applications by the West Midlands Gas Board, including the unloading of p.f.d. from railway tank wagons, where the freedom of both hands assists the operator when climbing and lifting the lid off the tank.

The powerful beam provided allows for complete examination of the interior of the tank. Because of its compactness and flexibility, the headpiece may also be used in place of a conventional handlamp for inspecting small and detailed parts of machinery.

A similar lamp, but constructed as a handlamp, is used in special locations including booster and exhauster houses. In these situations, where an explosive atmosphere may be present, a high degree of safety approval is also essential.

A PRACTICE MANOEUVRE

The team members have complete freedom in handling the injured man, with the lamp head fixed on their helmets . . .

Moving a casualty

During a practice, a rescue team (top) prepares to lower an injured man from the top of a treated water tank. In the lower picture the casualty, enclosed in a Neil Robertson stretcher, is lowered to ground level. Right: The team remove the victim by stretcher for medical attention.

PERSONAL NOTES

MR. W. K. HUTCHISON, C.B.E., Deputy Chairman, the Gas Council, has been elected President of the British Tar Confederation for 1961-62. Honorary Treasurer is MR. L. W. BLUNDELL, Controller of By-Products, North Thames Gas Board; Chairman of the Executive Board is MR. R. N. BRUCE, O.B.E., T.D., Chairman, South Eastern Gas Board; and Vice-Chairmen of the Executive Board are MR. C. LORD, Chairman, Lancashire Tar Distillers, Ltd., and LT.-COL. P. F. BENTON-JONES, O.B.E., Managing Director, United Coke & Chemicals Co. Ltd.

MR. P. DAWSON has been appointed Industrial Engineer (Administrative) to the North Western Gas Board; MR. K. B. GOULD is Heating Engineer (Gas); MR. S. POTTER is Technical Officer (Solid Fuel); MR. G. W. ROBERTSHAW is Development Engineer; and MR. H. H. THOMAS is Controller, Physical Testing Laboratories. These appointments follow the regrouping of commercial activities under a co-ordinating commercial manager, and are all within the department of the Technical Sales Manager at Board H.Q., MR. W. HOWELL.

MR. C. SMITH, who served the South Western Gas Board at Bath for 50 years, and for the past 13 years has been showroom supervisor there, has retired, as has MR. W. C. MANN, G.C.S. inspector at Exeter, who before going to Exeter in 1921 was with the Gas Light and Coke Company in London. At a Board meeting long service certificates and cheques were presented to them by the Deputy Chairman of the Board, Mr. A. H. Morland.

MR. J. SAMUELS, for six years a member of the Board, has been appointed Managing Director of Winston Electronics, Ltd. MR. W. A. BRIDGES continues as Chairman of the Board. Mr. Samuels joined Winston Electronics in its early days, and in 1956 was appointed purchasing director, then works director in charge of production, and two years ago sales director in charge of manufacturing sub-contracts.

COMING EVENTS

January 5.—LONDON AND SOUTHERN JUNIORS: Short paper meeting.

January 5.—SCOTTISH WESTERN JUNIORS: The King's Theatre, Glasgow. Theatre outing.

January 9.—MIDLAND JUNIORS: Staff Mess Room, West Midlands Gas Board, Birmingham District, Council House, Birmingham. 'Recent Developments in Retort House Practice at Saltley.'

January 9.—THE COMBUSTION ENGINEERING ASSOCIATION, NORTHERN REGION: Leeds. 'Liquid Petroleum Gas in Industry.'

MR. E. H. BADCOCK has been appointed Manager, Coal Department, and MR. G. SQUIRE has been appointed Manager, Building and Property Maintenance Department, of the North Thames Gas Board. Mr. Badcock succeeds MR. L. O. V. HAYWARD, who retired from the Board's service recently. Mr. Badcock was attached to the Coal Department early last year after having spent 30 years in the Board's Coke Department. Mr. Squire's appointment follows the retirement of MR. T. A. MARSHALL. Mr. Squire had been Mr. Marshall's deputy for the past ten years having joined the Board in 1946 as an assistant surveyor.

GAS COUNCIL ADVERTISING

WITH effect from April 1, 1962, Coleman, Prentis & Varley Ltd., will be handling the domestic advertising allocation of the Gas Council.

Technical and trade advertising will continue to be handled by C. P. Wakefield Ltd.

OBITUARY

MR. R. D. MACMILLAN, managing director of Controlled Heat & Air Ltd. (a member of the Incandescent Group of companies) has died at the age of 57. He joined Controlled Heat & Air Ltd. in 1933 as chief designer, and was appointed managing director in 1945. Mr. MacMillan concentrated mainly on the lower temperature phase of thermal engineering. He contributed substantially to the development of continuous tinplate printing machines, dust extraction in both wet and dry forms, related combustion equipment and heat exchangers, and critical control of high velocity convection in continuous static and mechanised forms.

MR. J. K. FISHER, works director of William Sugg & Co. Ltd., died at the Company's Westminster offices recently at the age of 54. Mr. Fisher had been associated with the Company for ten years and had been a member of the Board since July, 1959.

MR. A. GOLDTHORPE, a director of Hattersley & Davidson Ltd. and the Wormald Patent Locks Co. Ltd., has died at the age of 86. Mr. Goldthorpe had been associated with these Companies for over 50 years.

TRADE NOTES

RADIATION LTD., has agreed to acquire the domestic appliance interests in Australia of COATES & CO. LTD., and its subsidiary, RADCOATES (PTY.) LTD., subject to the approval of the shareholders of Coates & Co. Ltd. The terms are payment in cash for the book value of the net assets of the Appliance Division of Coates & Co. Ltd., including Radcoates (Pty.) Ltd., as at June 30, 1962. Coates & Co. Ltd., will continue with their other engineering activities which are outside the domestic appliance field.

THE MINERVA DETECTOR CO. LTD., Phelps House, 17, Heath Road, Twickenham, Middlesex, has recently moved its head office to Twickenham, in order to accommodate the larger sales and service facilities required by the increasing number of customers in Great Britain and Northern Ireland. To bring an extension of the business to the north of England, a Northern Region Sales and Service Centre has been opened in Bradford.

POWELL DUFFRYN HEATING LTD., recently introduced the new Janitor G.45 gas boiler which was featured at a special party to launch their new catalogue given by W. N. FROY & SONS LTD., King Street, W.6., at Tagg's Island. The Janitor was the centre-piece of a Powell Duffryn Heating equipment exhibition, which included a range of their radiators and other central heating installations.

GEORGE KENT LTD., Luton, Beds., received a party of visitors from Brooklands County Technical College. They were members of a course on management, conducted on behalf of the British Coking Industry Association. Their tour of the works included instrument assembly and calibration, Commander and Mark 3 instruments, and panel assembly departments.

FIELDEN ELECTRONICS LTD. Wythenshawe, Manchester 22, has changed the address of its London office. Owing to continued expansion and increase of sales the Southern Area office has moved to larger premises at 181-183 Earls Court Road, London S.W.5. Telephone: Freemantle 7866.

CANNON (HOLDINGS) LTD., has declared a dividend on the 5½% Redeemable Cumulative Preference shares of £1 each, at the rate of 5½% per annum, less tax, for the six months ending December 31, 1961, payable January 1, 1962.